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**Search Results - Record(s) 1 through 1 of 1 returned.**

1. Document ID: US 5874106 A

L23: Entry 1 of 1

File: USPT

Feb 23, 1999

US-PAT-NO: 5874106

DOCUMENT-IDENTIFIER: US 5874106 A

TITLE: Filled gelatin capsules

DATE-ISSUED: February 23, 1999

## INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Adesunloye; Adedotun Tony	Aurora	CO		
Stach; Paul Edward	Broomfield	CO		

US-CL-CURRENT: 424/456; 424/408, 514/962

Full	Title	Citation	Front	Review	Classification	Date	Reference
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Image	Draw Desc	Image
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Term	Documents
BENZOIC.DWPI,TDBD,EPAB,JPAB,USPT,PGPB.	64709
BENZOICS.DWPI,TDBD,EPAB,JPAB,USPT,PGPB.	7
ACID[USPT,PGPB]	552538
ACIDS[USPT,PGPB]	313052
(10 AND (BENZOIC ADJ (ACID[CLM]))) USPT,PGPB,JPAB,EPAB,DWPI,TDBD.	1

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10

Documents, starting with Document: 1

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Art Unit: 1711

Taubitz does not mention <sup>as well as</sup> the (claimed) particle size of metallic particles and mineral particles/average diameter and average aspect ratio of fibers. *WKS*

Levine discloses that a preferred metallic pigment should have a diameter of 10-20

microns (see Levine, col. 5, lines 30-34).

Kohara discloses that the average diameter of a fibrous filler is 0.1 to 100 micrometers, and the length is from 5 micrometers to 20 millimeters. Average <sup>particle</sup> ~~aspect~~ diameter of powdery filler is 0.01 micrometer to 1 millimeter (see Kohara, col. 4, lines 27-38).

Clark discloses thermoplastic compositions having a speckled surface appearance; This appearance results <sup>from</sup> ~~from~~ the use of nondispersing pigments (abstract; col. 10, lines 1-2). It is preferred that these pigments possess a large aspect ratio, <sup>greater</sup> ~~greater~~ than about 20 (col. 10, lines 33-

38).

Based on the teachings of Levine, Kohara and Clark, it is the examiner's position that it would have been obvious to one ordinary skill in the art to use in the composition of Taubitz;

metallic particles of claimed particle size, fibers of claimed average diameter and average aspect ratio and <sup>metallic</sup> ~~metallic~~ particles of claimed average particle size in order to produce products having bright reflective appearance, enhanced hiding power, improved crack resistance and moldability

as well as better speckled appearance.

4. Any inquiry concerning this communication or earlier communications from the

examiner should be directed to U.K. Rajguru whose telephone number is (703) 308-3224. The

examiner can normally be reached on Monday - Friday from 9:30 am to 6:00 pm.

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**Search Results - Record(s) 1 through 10 of 11 returned.****1. Document ID: US 6211268 B1**

L17: Entry 1 of 11

File: USPT

Apr 3, 2001

US-PAT-NO: 6211268

DOCUMENT-IDENTIFIER: US 6211268 B1

TITLE: Polyoxymethylene resin composition

DATE-ISSUED: April 3, 2001

## INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Matsumura; Takatoshi	Yokkaichi			JPX
Nishizawa; Chiharu	Yokkaichi			JPX
Mimura; Hiroshi	Yokkaichi			JPX
Yada; Hiroshi	Yokkaichi			JPX
Kurashige; Kazuo	Yokkaichi			JPX

US-CL-CURRENT: 524/100; 524/275, 524/377, 524/378, 524/384, 524/394, 524/400

Full	Title	Citation	Front	Review	Classification	Date	Reference	Claims	FIGS	Draw Desc	Image
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**2. Document ID: US 6077908 A**

L17: Entry 2 of 11

File: USPT

Jun 20, 2000

US-PAT-NO: 6077908

DOCUMENT-IDENTIFIER: US 6077908 A

TITLE: Polyoxymethylene resin composition

DATE-ISSUED: June 20, 2000

## INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Yahiro; Shyuzi	Kurashiki			JPX

US-CL-CURRENT: 525/218; 525/282, 528/310, 528/322

Full	Title	Citation	Front	Review	Classification	Date	Reference	Claims	FIGS	Draw Desc	Image
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**3. Document ID: US 5310823 A**

L17: Entry 3 of 11

File: USPT

May 10, 1994

Art Unit: 1711

1. A request for reconsideration has been filed on May 23, 2000 (paper no. 4).
2. The text of those sections of Title 35, U.S. Code not included in this action can be found in a prior Office action.

Claims 1-12 are rejected under 35 U.S.C. 103(a) as being unpatentable over Morris (USP 3919143) in view of EP 811659 and Yogishita et al (USP 4931509).

This rejection is incorporated hereby reference from prior office action paper no. 3.

Applicants state that (page 3, paragraphs 1-3) Morris uses polyvalent carboxylic acid salts and not acids as required by instant claim 1. This is not persuasive when Morris does specifically state in col. 5, lines 12-145 that on polyvalent carboxylic acid is used.

Examples in prior art are considered illustrative and not limitative.

Therefore applicants' other argument that "Morris presents only one example as an acid" is not persuasive.

Applicants comment (p. 4, paragraph 5), that EP '659 discloses that

hydrotalcite does not improve compression set. This comment is true but relevant

21 has been

US-PAT-NO: 5310823  
DOCUMENT-IDENTIFIER: US 5310823 A  
TITLE: Polyacetal resin composition  
DATE-ISSUED: May 10, 1994

## INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Kunitomi; Masaki	Nagoya			JPX
Ishii; Hiromitsu	Nagoya			JPX
Yamamoto; Yoshiyuki	Suzuka			JPX

US-CL-CURRENT: 525/400; 524/155, 524/157, 524/158, 524/161, 524/282, 525/80,  
525/90, 525/93

Full	Title	Citation	Front	Review	Classification	Date	Reference
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RVOC	Draw Desc	Image
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## 4. Document ID: US 4987174 A

L17: Entry 4 of 11

File: USPT

Jan 22, 1991

US-PAT-NO: 4987174  
DOCUMENT-IDENTIFIER: US 4987174 A

TITLE: Low gloss polyacetal resin

DATE-ISSUED: January 22, 1991

## INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Ying; Edwina	Bridgewater	NJ		
Hayes; Conrad G.	Plainfield	NJ		

US-CL-CURRENT: 524/444; 524/450, 524/593

Full	Title	Citation	Front	Review	Classification	Date	Reference
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RVOC	Draw Desc	Image
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## 5. Document ID: US 4855365 A

L17: Entry 5 of 11

File: USPT

Aug 8, 1989

Art Unit: 1711

will the statutory period for reply expire later than SIX MONTHS from the mailing date of this final action.

4. Any inquiry concerning this communication or earlier communications from

the examiner should be directed to U.K. Rajguru whose telephone number is (703) 308-3224. The examiner can normally be reached on Monday - Friday from 9:30 am to 6:00 pm.

If attempts to reach the examiner by telephone are unsuccessful, the examiner's supervisor, Jim Seidleck, can be reached on (703) 308-2462. The fax

phone number for the organization where this application or proceeding is assigned is (703) 305-3599.

Any inquiry of a general nature or relating to the status of this application or

proceeding should be directed to the receptionist whose telephone number is (703)

308-0661.

U.K. Rajguru/om  
August 18, 2000

US-PAT-NO: 4855365  
DOCUMENT-IDENTIFIER: US 4855365 A

TITLE: Poly-.beta.-alanine compound, a process for producing the same and a polyacetal resin composition containing a poly-.beta.-alanine compound

DATE-ISSUED: August 8, 1989

## INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Yamamoto; Fumihiko	Kawasaki			JPX
Misumi; Teruyuki	Yokohama			JPX

US-CL-CURRENT: 525/401; 525/393, 525/402, 525/427, 525/472, 526/192, 526/210,  
526/212, 528/310, 528/328, 528/332

Full	Title	Citation	Front	Review	Classification	Date	Reference
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## 6. Document ID: JP 2000169667 A

L17: Entry 6 of 11

File: JPAB

Jun 20, 2000

PUB-NO: JP02000169667A

DOCUMENT-IDENTIFIER: JP 2000169667 A

TITLE: REINFORCED POLYACETAL RESIN COMPOSITION AND INJECTION MOLDED PRODUCT

PUBN-DATE: June 20, 2000

## INVENTOR-INFORMATION:

NAME	COUNTRY
YAMANAKA, TORU	
MAKABE, YOSHIKI	
NISHIMURA, TORU	

INT-CL (IPC): C08L 59/00; B29C 45/00; C08K 3/02; C08K 3/34; C08K 5/13; C08K 5/17;  
C08K 5/3492; C08K 5/521

Full	Title	Citation	Front	Review	Classification	Date	Reference
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## 7. Document ID: JP 11100486 A

L17: Entry 7 of 11

File: DWPI

Apr 13, 1999

pages  
Thanks

Jeffrey A. Furman, Esq.

1. A request for extension of time (paper no. 8) and an amendment (paper no. 9) have been filed on May 11, 2000.

2. Applicant's election with traverse of claims 1-9 ~~are~~ in Paper No. 9 is acknowledged. The traversal is on the ground(s) that it would not constitute an undue burden to search all the subject matter as claimed. This is not found persuasive because the applicants have made only a

conclusionary statement without any support for it. There is actually an undue burden for search for non-elected claim 10.

The requirement is still deemed proper and is therefore made FINAL.

3. Rejection of claim <sup>1</sup>under 35 U.S.C. 112, second paragraph (see section 3, page 3 of office action paper no. 5) is now withdrawn.

4. The text of those sections of Title 35, U.S. Code not included in this action can be found in a prior Office action.

Claims 1-9 and (newly added) 11-14 are rejected under 35 U.S.C. 103(a) as being

unpatentable over Schlumpf (Canadian Patent 2016447) in view of Shaw et al (USP 4643940), Hosoda et al (USP 3812225) and Figlii et al (USP 4680318).

This rejection (also to be applied to new claims 11-14) is incorporated here by reference

from section 4, pages 3-5, of prior office, paper no. 5.

Applicant's arguments filed May 11, 2000 (paper no. 9) have been fully considered but

they are not persuasive.



DERWENT-ACC-NO: 1999-296594  
DERWENT-WEEK: 199928  
COPYRIGHT 2001 DERWENT INFORMATION LTD

TITLE: Polyoxymethylene resin composition - with good heat stability and moldability

PRIORITY-DATA: 1997JP-0263431 (September 29, 1997)

PATENT-FAMILY:

PUB-NO	PUB-DATE	LANGUAGE	PAGES	MAIN-IPC
JP 11100486 A	April 13, 1999		008	C08L059/00

INT-CL (IPC): C08K 5/15; C08K 5/29; C08L 59/00

Full	Title	Citation	Front	Review	Classification	Date	Reference
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FORMC	Draw Desc	Image
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8. Document ID: JP 07188514 A

L17: Entry 8 of 11

File: DWPI

Jul 25, 1995

DERWENT-ACC-NO: 1995-290564  
DERWENT-WEEK: 199538  
COPYRIGHT 2001 DERWENT INFORMATION LTD

TITLE: Polyacetal resin compsn. used for moulding car electrical parts - comprising hindered phenol type antioxidant and formaldehyde scavenger

PRIORITY-DATA: 1993JP-0333738 (December 27, 1993)

PATENT-FAMILY:

PUB-NO	PUB-DATE	LANGUAGE	PAGES	MAIN-IPC
JP 07188514 A	July 25, 1995		008	C08L059/00

INT-CL (IPC): C08K 5/13; C08L 59/00

Full	Title	Citation	Front	Review	Classification	Date	Reference
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FORMC	Draw Desc	Image
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9. Document ID: JP 06256621 A

L17: Entry 9 of 11

File: DWPI

Sep 13, 1994

Art Unit: 1711

Applicant's arguments do not comply with 37 CFR 1.111(c) because they do not clearly point out the patentable novelty which he or she thinks the claims present in view of the state of the art disclosed by the references cited or the objections made. Further, they do not show how the amendments avoid such references or objections.

On page 6, paragraph 2 of above response paper no. 9, the applicants argue that Schlumpf teaches away from claimed invention because Schlumpf discloses solids at least 92.1%. While there is a difference of only 0.1% (between claimed 92.0% and disclosed 92.1%), this difference fails to distinguish patentably the instant invention from the prior art, particularly because the applicants have not established the criticality of the upper limit viz. 92.0%.

Applicants' further argue in the next paragraph on the same page, that Schlumpf does not teach claimed particle size distribution. <sup>his</sup> argument is not persuasive. On page 9, Schlumpf discloses a calcium carbonate of a mean statistical particle diameter of 0.5 - 50 microns. This range overlaps that of calcium carbonate (viz. 0.3 to 18 microns) in example 1 in instant specification page 14. Furthermore, <sup>also</sup> the applicants have not shown any criticality of having the only the claimed particle size distribution.

On page 6, paragraph 4 of same paper, the applicants state that apparatus of Schlumpf cannot degas the composition and therefore Schlumpf teaches away ~~from~~ <sup>from</sup> inclusion of a blowing agent. <sup>Whether the apparatus can or cannot degas the composition has no influence at all. It is the effect the composition has on the process that matters. Claims are rendered obvious by the prior art.</sup>

DERWENT-ACC-NO: 1994-330256  
DERWENT-WEEK: 199441  
COPYRIGHT 2001 DERWENT INFORMATION LTD

TITLE: Polyoxymethylene resin compsn. for moulding e.g. car parts - includes a hypophosphite cpd., hindered phenol and amine cpds. and a formaldehyde scavenger

PRIORITY-DATA: 1993JP-0043688 (March 4, 1993)

PATENT-FAMILY:

PUB-NO	PUB-DATE	LANGUAGE	PAGES	MAIN-IPC
JP 06256621 A	September 13, 1994		008	C08L059/00

INT-CL (IPC): C08K 5/52; C08L 59/00

Full	Title	Citation	Front	Review	Classification	Date	Reference
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RMWD	Draw Desc	Clip Img	Image
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10. Document ID: JP 3168639 B2, JP 05125255 A

L17: Entry 10 of 11

File: DWPI

May 21, 2001

DERWENT-ACC-NO: 1993-200634  
DERWENT-WEEK: 200130  
COPYRIGHT 2001 DERWENT INFORMATION LTD

TITLE: Polyoxymethylene resin compsn. for reduced formaldehyde odour and mould deposits - comprises polyoxymethylene resin contg. oxazoline and/or oxadiazoline cpd. hindered phenol and amine cpds. and formaldehyde scavenger for heat resistance

PRIORITY-DATA: 1991JP-0291568 (November 7, 1991)

PATENT-FAMILY:

PUB-NO	PUB-DATE	LANGUAGE	PAGES	MAIN-IPC
JP 3168639 B2	May 21, 2001		010	C08L059/00
JP 05125255 A	May 21, 1993		010	C08L059/00

INT-CL (IPC): C08K 5/13; C08K 5/18; C08K 5/35; C08L 59/00

Full	Title	Citation	Front	Review	Classification	Date	Reference
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RMWD	Draw Desc	Clip Img	Image
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Term

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11

Display

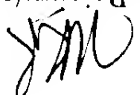
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Documents, starting with Document.

11

Art Unit: 1711

Any inquiry of a general nature or relating to the status of this application or proceeding should be directed to the receptionist whose telephone number is (703) 308-0661.

  
U.K. Rajguru/om  
August 14, 2000

<u>DB Name</u>	<u>Query</u>	<u>Hit Count</u>	<u>Set Name</u>
USPT,PGPB,JPAB,EPAB,DWPI,TDBD	(polyacetal resin or polyoxymethylene resin) and (anthranilic acid or 2 adj amino benzoic acid or 4 adj amino benzoic acid)	21	<a href="#">L19</a>
USPT,PGPB,JPAB,EPAB,DWPI,TDBD	(polyacetal resin or polyoxymethylene resin) and (anthranilic acid or 4 adj amino benzoic acid) near15 low formaldehyde	0	<a href="#">L18</a>
USPT,PGPB,JPAB,EPAB,DWPI,TDBD	(anthranilic acid or 4 adj amino benzoic acid) near15 low formaldehyde	0	<a href="#">L17</a>
USPT,PGPB,JPAB,EPAB,DWPI,TDBD	(anthranilic acid or 2 adj amino benzoic acid) near15 low formaldehyde	0	<a href="#">L16</a>
USPT,PGPB,JPAB,EPAB,DWPI,TDBD	(anthranilic acid or 2 adj amino benzoic acid) near5 low formaldehyde	0	<a href="#">L15</a>
USPT,PGPB,JPAB,EPAB,DWPI,TDBD	(anthranilic acid or 2 adj amino benzoic acid) and low formaldehyde	8	<a href="#">L14</a>
USPT,PGPB,JPAB,EPAB,DWPI,TDBD	11 and low formaldehyde	7	<a href="#">L13</a>
USPT,PGPB,JPAB,EPAB,DWPI,TDBD	11 and composit\$3 and article and (mould?? or mold??)and low formaldehyde	0	<a href="#">L12</a>
USPT,PGPB,JPAB,EPAB,DWPI,TDBD	11 and composit\$3 and article and (mould?? or mold??)	37	<a href="#">L11</a>
USPT,PGPB,JPAB,EPAB,DWPI,TDBD	11 and composit\$3 and article	62	<a href="#">L10</a>
USPT,PGPB,JPAB,EPAB,DWPI,TDBD	11 and composit\$3	164	<a href="#">L9</a>
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USPT,PGPB,JPAB,EPAB,DWPI,TDBD	polyacetal and 4 adj amino benzoic acid [ab]	1	<a href="#">L7</a>
USPT,PGPB,JPAB,EPAB,DWPI,TDBD	polyacetal and 4 adj amino benzoic acid [ti]	0	<a href="#">L6</a>
USPT,PGPB,JPAB,EPAB,DWPI,TDBD	polyacetal and 4 adj amino benzoic acid	21	<a href="#">L5</a>
USPT,PGPB,JPAB,EPAB,DWPI,TDBD	polyacetal and (anthranilic acid or 4 adj amino benzoic acid) [clm]	1	<a href="#">L4</a>
USPT,PGPB,JPAB,EPAB,DWPI,TDBD	polyacetal and (anthranilic acid or 4 adj amino benzoic acid) [ab]	3	<a href="#">L3</a>
USPT,PGPB,JPAB,EPAB,DWPI,TDBD	polyacetal and (anthranilic acid or 4 adj amino benzoic acid) [ti]	1	<a href="#">L2</a>
USPT,PGPB,JPAB,EPAB,DWPI,TDBD	polyacetal and (anthranilic acid or 4 adj amino benzoic acid)	260	<a href="#">L1</a>

Art Unit: 1711

variants. Should applicant traverse on the ground that the species are not patentably distinct, applicant should submit evidence or identify such evidence now of record showing the species to be obvious variants or clearly admit on the record that this is the case. In either instance, if the examiner finds one of the inventions anticipated by the prior art, the evidence or admission may be used in a rejection under 35 U.S.C. 103(a) of the other invention.

Because these inventions are distinct for the reasons given above and have acquired a separate status in the art because of their recognized divergent subject matter, restriction for examination purposes as indicated is proper.

Because these inventions are distinct for the reasons given above and the search required for Group I or II is not required for Group II, restriction for examination purposes as indicated is proper.

A telephone call was made to Attorney Arnold Turk on August 9, 2000 to request an oral election to the above restriction requirement, but did not result in an election being made.

Any inquiry concerning this communication or earlier communications from the examiner should be directed to U.K. Rajguru whose telephone number is (703) 308-3224. The examiner can normally be reached on Monday - Friday from 9:30 am to 6:00 pm.

If attempts to reach the examiner by telephone are unsuccessful, the examiner's supervisor, Jim Seidleck, can be reached on (703) 308-2462. The fax phone number for the organization where this application or proceeding is assigned is (703) 305-3599.

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L15: Entry 2 of 2

File: DWPI

DERWENT-ACC-NO: 1968-12294Q  
DERWENT-WEEK: 196800  
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09/852383

TITLE: Polyoxymethylene stabilisation with aliphatic or

PATENT-ASSIGNEE:

ASSIGNEE

CODE

MITR

MITR

MITSUBISHI RAYON CO LTD

MITR

PRIORITY-DATA: 1964JP-0025050 (May 4, 1964)

PATENT-FAMILY:

PUB-NO

PUB-DATE

LANGUAGE

PAGES

MAIN-IPC

JP 68020308 B

000

ABSTRACTED-PUB-NO: JP68020308B

BASIC-ABSTRACT:

Polyoxymethylene of high mol. wt. having at least 1 terminal OH gp. is stabilised with aliphatic or aromatic dicarboxylic acid imide or o-sulphobenzimide, opt. ring substd., using 3-20% aqs. NH4OH as reaction medium.

Pref. 10 parts polyoxymethylene, 10-1000 parts 3-20% NH4OH and 3-0.1 parts stabiliser heated and stirred at 50-100 deg.C, esp. 80-90 deg.C for 0.5-5 hr. in sealed tube, opt. under N2. Suitable stabilisers include phthalimide and 3-chloro-phthalimide, malonimide, succinimide, glutamimide, adipimide and o-sulphobenzimide opt. ring substd. by alkyl, alkenyl, cycloalkyl, allyl, amino, acylamine or COOH. Polyoxymethylene is pref. reduced to particle size below 50 mesh.

TITLE-TERMS : POLYOXYMETHYLENE STABILISED ALIPHATIC

DERWENT-CLASS: A00

CPI-CODES: A05-H02; A08-A01; A09-A01; A10-E01;

Multipunch Codes: 01- 080 138 180 231 250 273 31- 329 331 359 393 541 546 681 688  
720 721

cal oligomerization. Examples of structure suitable for E include straight chain, branched chain or cyclic alkyl-ene, arylene, aralkylene, oxy, oxo, hydroxy, thio, sulfo-nyl, sulfoxy, imino, sulfonamido, acylimino, acyloxy, urethaneylene, ureylene, and combinations thereof. E is preferably alkylene.

As illustrated in Formulas I-III, a compound can contain a plurality of Z groups each bonded to the fluorochemical oligomeric portion through any of R<sub>1</sub>, R<sub>2</sub>, R<sub>3</sub>, Q, X and/or E. In most embodiments, it is preferred that there be 1 group Z in a compound, preferred being bonded to the fluorochemical oligomer portion through group E in a compound of Formulas I or II, or through group X in a compound of Formulas III. These embodiments are discussed in detail below in connection with Schemes I and II below. However, in an instance wherein the group R is a mono, di, or polyvalent polymeric chain (e.g., as set forth in Examples 97-100 below), the number of Z groups is preferably greater than 1.

Compositions of the invention can be prepared as shown in Schemes I and II below, wherein a, b, q, R<sub>1</sub>, R<sub>2</sub>, Q, R<sub>3</sub>, X, E, R<sub>4</sub> and R are as defined above, and R<sub>5</sub>, R<sub>6</sub>, Q, R<sub>7</sub>, Y and L generally correspond to R<sub>1</sub>, R<sub>2</sub>, Q, R<sub>3</sub>, X, E, and R, respectively, absent the proviso that at least one thereof has an unsatisfied valence, and with the proviso that at least one thereof possesses a functional group capable of reacting with an isocyanate or capable of being converted or further elaborated into such a functional group. Products other than those shown in the schemes will be produced by the various steps. The schemes show only the major products of the various steps.





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ODOR.DWPI,TDBD,EPAB,JPAB,USPT,PGPB.	46240
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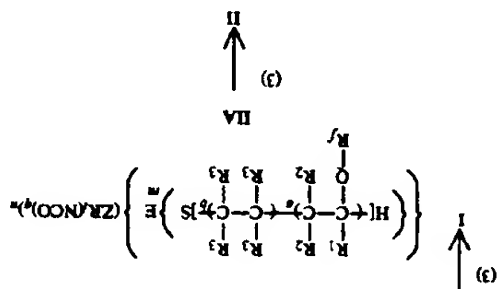
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114 and odor reducing

Refine Search:

[Clear](#)**Search History****Today's Date: 12/19/2001**

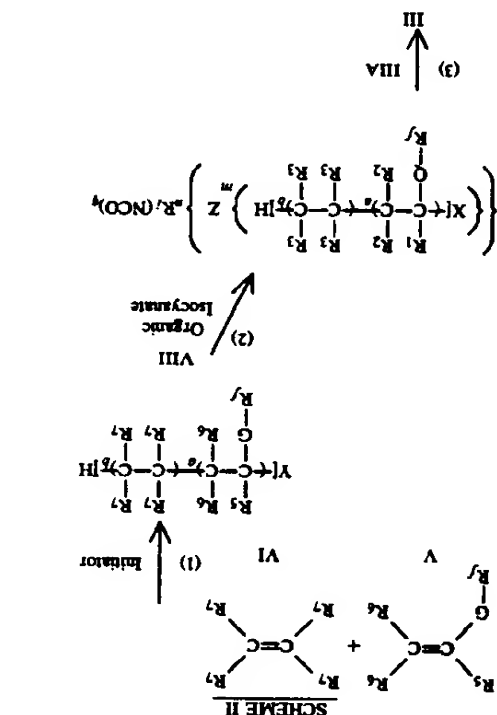
<u>DB Name</u>	<u>Query</u>	<u>Hit Count</u>	<u>Set Name</u>
USPT,PGPB,JPAB,EPAB,DWPI,TDBD	114 and odor reducing	1	L24
USPT,PGPB,JPAB,EPAB,DWPI,TDBD	114 and composit\$3 and molded article	8	L23
USPT,PGPB,JPAB,EPAB,DWPI,TDBD	114 and composit\$3	205	L22
USPT,PGPB,JPAB,EPAB,DWPI,TDBD	succinimide same (polyacetal or polyoxymethylene)	3	L21



**SCHEME I**

In step (1) of Scheme 1, a fluorocemical monomer of Formula V is oligomerized in the presence of a free-radical initiator, an end-capping agent or the formula L(SH)<sup>m</sup>, and optionally a non-fluorinated comonomer of Formula VI. In Scheme II, the monomers are the same, the end-capping agent is optionally present, and the initiator is preferably a functional initiator.

Compounds of Formula V and methods for the preparation thereof are known and disclosed, e.g., in U.S. Pat. Nos. 2,803,615 (Abibrecht et al.) and 2,641,573 (Abibrecht et al.) which disclosures are incorporated herein by reference. Examples of such compounds include general classes of fluorocemical olefins such as acrylates, methacrylates, vinyl ethers, and allyl compounds containing fluorinated sulfonyl groups, acrylates or methacrylates derived from fluorocemical telomer alcohols, fluorocemical thiols, and the like. Preferred compounds of Formula V include such as N-methyl perfluorooctanesulfonamidoethyl acrylate, N-ethyl perfluorooctanesulfonamidoethyl acrylate, N-nonyl perfluorooctanesulfonamidoethyl acrylate, N-octyl perfluorooctanesulfonamidoethyl acrylate, N-hexyl perfluorooctanesulfonamidoethyl acrylate, N-butyl perfluorooctanesulfonamidoethyl acrylate, N-propyl perfluorooctanesulfonamidoethyl acrylate, N-methylperfluorohexylsulfonamidoethyl acrylate, the reaction product of isocyanatoethyl methacry-



latic acid and N-methylperfluorooctanesulfonamidoethyl alcohol, perfluorooctyl acrylate, N-methyl perfluorooctanesulfonamidoethyl acrylate, and others such as perfluorocyclohexyl acrylate, and tetrameric hexafluoropropyleneoxide dihydroacrylate.

Compounds of Formula VI are also well known and generally commercially available. Examples of such compounds include general classes of ethylenic compounds capable of free-radical polymerization, such as lower olefinic hydrocarbons, optionally halogenated, such as ethylene, propylene, isobutene, 3-chloro-2-isobutene, butadiene, isoprene, chloro and dichlorobutadienes, fluoro and difluorobutadienes, 2,5-dimethyl-1,5-hexadiene; vinyl, allyl or vinylidene halides such as vinyl or vinylidene chloride, vinyl or vinylidene fluoride, allyl bromide, allyl chloride, methyl-1-chloride; styrene and its derivatives such as vinyltoluene,  $\alpha$ -methylstyrene,  $\alpha$ -cyanomethylstyrene, divinylbenzene, N-vinylcarbazole; vinyl esters such as vinyl acetate, vinyl propionate, vinyl isobutyrate, vinyl succinate, vinyl itaconate, divinylfumarate, allyl esters such as allyl acetate and allyl heptanoate; alkylvinyl or alkylallyl ethers such as cetyl vinyl ether, dodecyl vinyl ether, isobutyl vinyl ether, ethyl vinyl ether, 2-chloroethyl vinyl ether, tetraallyloxyethane; vinyl alkyl ketones such as vinyl methyl ketone; unsaturated acids such as acrylic,  $\alpha$ -chloro acrylic,  $\alpha$ -fluoro acrylic, crotonic, maleic, fumaric, itaconic, and citraconic acids, and amides and esters thereof such as dimethyl maleate, ethyl crotonate, acid methyl maleate, acid butyl itaconate, and vinyl, allyl, methyl, ethyl, butyl, isobutyl, hexyl, 2-ethylhexyl, chlorohexyl, octyl, lauryl, or stearyl acrylates and methacrylates; olefinic silanes such as vinyltrichlorosilane, vinyltrimethoxysilane, vinyltriethoxysilane, and methacryloyloxypropyl trimethoxysilane; nitriles such as acrylonitrile, methacrylonitrile, 2-chloroacrylonitrile, 2-cyanoethylacrylate, methylacrylate, vinylidene cyanide, (tri- $\alpha$ -cyloilo)-beta-cyanoacrylate; special acrylates such as butanediol di-methacrylate, dicyclopentyl acrylate, ethoxydiol dimethacrylate, trimethacrylate, isobornyl acrylate and methacrylate, A dimethacrylate, isobornyl acrylate and methacrylate; acrylamides and methacrylamides; mono or di (meth)acrylates of glycols or polyalkylene glycols such as ethylene glycol dimethacrylate, triethylene glycol acrylate, mono, di, and polyacrylates and methacrylates of methoxypolyethylene glycols and polyethylene glycols of various molecular weights (available as CARBOWAX™), block copolymers of ethylene oxide and propylene oxide endcapped by hydroxy-

USPT,PGPB,JPAB,EPAB,DWPI,TDBD	succinimide near15 (polyacetal or polyoxymethylene)	0	<a href="#">L20</a>
USPT,PGPB,JPAB,EPAB,DWPI,TDBD	succinimide near5 (polyacetal or polyoxymethylene)	0	<a href="#">L19</a>
USPT,PGPB,JPAB,EPAB,DWPI,TDBD	114 and formaldehyde scavenger	0	<a href="#">L18</a>
USPT,PGPB,JPAB,EPAB,DWPI,TDBD	succinimide and (polyacetal or polyoxymethylene) [clm]	14	<a href="#">L17</a>
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USPT,PGPB,JPAB,EPAB,DWPI,TDBD	succinimide and (polyacetal or polyoxymethylene) [ti]	2	<a href="#">L15</a>
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USPT,PGPB	succinimide near formaldehyde scavenger	0	<a href="#">L5</a>
USPT,PGPB	succinimide and formaldehyde scavenger	11	<a href="#">L4</a>
USPT,PGPB	succinimide and 3406223 [pn]	0	<a href="#">L3</a>
USPT,PGPB	succinimide and 5603927 [pn]	0	<a href="#">L2</a>
USPT,PGPB	succinimide and 5866671 [pn]	0	<a href="#">L1</a>

[illegible]

<u>DB Name</u>	<u>Query</u>	<u>Hit Count</u>	<u>Set Name</u>
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USPT,PGPB,JPAB,EPAB,DWPI,TDBD	110 and 113	25	<a href="#">L14</a>
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User Name: w

User Phone:

Workstation Id: GPRBURGM

Printer Id: gbgmptr

Date: Thu Nov 12, 1998

Time: 12:40:30

# Job # 8753

<u>Patent ID</u>	<u>Document Not Available</u>	<u>Pages Not Available</u>	<u>150 dpi Pages</u>	<u>Unscanned Pages</u>	<u>Total Pages Printed</u>
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**WEST**

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**Search Results - Record(s) 1 through 2 of 2 returned.****1. Document ID: WO 200059993 A1**

L15: Entry 1 of 2

File: DWPI

Oct 12, 2000

DERWENT-ACC-NO: 2001-024449

DERWENT-WEEK: 200103

COPYRIGHT 2001 DERWENT INFORMATION LTD

TITLE: New polyacetal resin composition useful in molded articles comprises at least one odor reducing additive e.g. tris(hydroxymethyl)aminomethane or ethyl p-aminobenzoate

INVENTOR: KASSAL, R J; MORI, H ; SHINOHARA, K

PRIORITY-DATA: 1999US-0287432 (April 7, 1999)

PATENT-FAMILY:

PUB-NO	PUB-DATE	LANGUAGE	PAGES	MAIN-IPC
WO 200059993 A1	October 12, 2000	E	018	C08K005/00

INT-CL (IPC): C08K 5/00; C08L 59/00; C08K 5/3445; C08K 5/17; C08K 5/00

Full	Title	Citation	Front	Review	Classification	Date	Reference	Claims	FORM	Draw Data	Image
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**2. Document ID: JP 68020308 B**

L15: Entry 2 of 2

File: DWPI

DERWENT-ACC-NO: 1968-12294Q

DERWENT-WEEK: 196800

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TITLE: Polyoxymethylene stabilisation with aliphatic or

PRIORITY-DATA: 1964JP-0025050 (May 4, 1964)

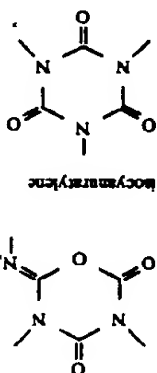
PATENT-FAMILY:

PUB-NO	PUB-DATE	LANGUAGE	PAGES	MAIN-IPC
JP 68020308 B			000	

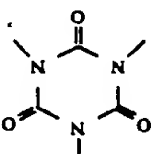
Full	Title	Citation	Front	Review	Classification	Date	Reference	Claims	FORM	Draw Data	Image
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Generate Collection

10  
-continued



10 bicyanuric triazine

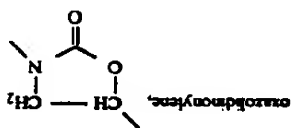


and the like.

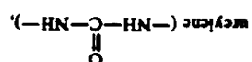
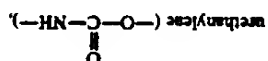
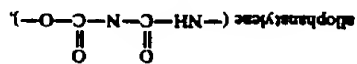
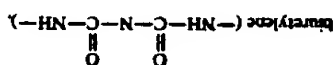
A fluorinated compound of the invention also contains a group that can impart soft hand, stain release, water repellency, or a durable property when the compound is applied to a fibrous substrate. A group that can impart stain release is comprised by the fluorochromic portion. In particular, any one or more of  $R_1, R_2, R_3, E$ , or  $X$  can comprise such a group. It is preferred that groups that can impart soft hand, water-repellency, or a durable property be comprised by the organic moiety  $R$ . Such a group, however, can alternatively be comprised by the fluorochromic oligomeric portion. In particular, with reference to Formulas I-III, any one or more of the substituents  $Q, R_1, R_2, R_3, R, E$  or  $X$  can comprise such a group.

Any property that can be imparted through the use of fluorochromic compounds on fibrous substrates can be imparted to the compositions of the invention, and any such property can be rendered a durable property through the use of the compositions of the invention. Durability can be achieved by any of a number of mechanisms of interaction between the fluorinated compound and a substrate. For example, interactions such as physical entanglement, or chemical interaction such as covalent bonding by way of nucleophilic, electrophilic, ionic, free radical or like reactions between the fluorinated compound and the substrate, or ionic bonding, can cause durability. Wool, leather, paper, cotton, and nylon variously comprise hydroxyl, amino, carboxyl, and carboxamido groups. Other fabrics such as some non-wovens are essentially non-functional but comprise groups in  $R$  be substantially unreactive to isocyanates under conditions conventionally employed to react a nucleophile with an isocyanate.

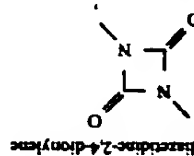
The organic moiety  $R$  can also contain isocyanate-derived moieties such as those described above in connection with linking group  $Z$ . Further,  $R$  can comprise isocyanate-derived moieties that are formed by the reaction of two or more isocyanates with one another, such as carbodiimidylenes ( $-N=C=N-$ ).



15 guanidinylenes ( $-N-C(=NH)-NH-$ )



20 acetylurea ( $-CONH-$ )



25 4,6-dioxobenzodiazepine-1,3,5-oxadiazine



**WEST**

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L4: Entry 1 of 11

File: PGPB

Dec 13, 2001

PGPUB-DOCUMENT-NUMBER: 20010051679  
PGPUB-FILING-TYPE: new  
DOCUMENT-IDENTIFIER: US 20010051679 A1

TITLE: Amino resin mixture for producing foils and edgings with reduced formaldehyde elimination

PUBLICATION-DATE: December 13, 2001

## INVENTOR-INFORMATION:

NAME	CITY	STATE	COUNTRY	RULE-47
Scholl, Frank	Bad Homburg		DE	
Wonner, Johann	Rodgau		DE	
Hobisch, Gerald	Graz		AT	

APPL-NO: 09/ 847478  
DATE FILED: May 2, 2001

## FOREIGN-APPL-PRIORITY-DATA:

COUNTRY	APPL-NO	DOC-ID	APPL-DATE
DE	100 21 849.0	2000DE-100 21 849.0	May 5, 2000

INT-CL: [07] C08K 3/20, C08F 283/04, C08K 5/36  
US-CL-PUBLISHED: 524/211; 524/501, 525/453  
US-CL-CURRENT: 524/211; 524/501, 525/453

## ABSTRACT:

Amino resin mixture for producing foils and edgings with reduced formaldehyde elimination Aqueous amino resin compositions comprising amino resins A, formaldehyde-binding additives (auxiliaries) B, which may include hydroxyl-containing polyurethanes B13, acrylic resins C, if desired, in the form of aqueous dispersions, and water, and their use as impregnating compositions for paper for the purpose of producing finished foils and edgings.

## FIELD OF THE INVENTION

{0001} The present invention relates to aqueous, heat-curable amino resin mixtures based on etherified amino resins and formaldehyde-binding auxiliaries and to their use for impregnating papers and cardboard.

## BACKGROUND OF THE INVENTION

{0002} Surfaces and narrow faces of woodbase materials are coated using coated foils (finished foils) and, respectively, impregnated cardboard (Kunststoff-Handbuch Vol. 10 Duroplaste [Thermosets], Hanser-Verlag, 2nd Ed. 1988, p. 462 f., pp. 477 to 479). For the production of finished foils (to coat surfaces) and finished edgings (to coat narrow faces, of boards in particular), absorbent papers are impregnated with solutions or dispersions of (thermosetting) amino resins such as urea-formaldehyde and/or melamine formaldehyde resins, for example, alone or in combination with dispersions of thermoplastics such as acrylic dispersions or styrene-acrylic dispersions, dried in a stream of hot air and simultaneously cured, and then coated.

1. A request for extension of time (paper no. 8) and an amendment (paper no. 9) have been filed on May 11, 2000.
2. Applicant's election with traverse of claims 1-9 are in Paper No. 9 is acknowledged. The traversal is on the ground(s) that it would not constitute an undue burden to search all the subject matter as claimed. This is not found persuasive because the applicants have made only a conclusory statement without any support for it. There is actually an undue burden for search for non-elected claim 10.
- The requirement is still deemed proper and is therefore made FINAL.
3. Rejection of claim under 35 U.S.C. 112, second paragraph (see section 3, page 3 of office action paper no. 5) is now withdrawn.
4. The text of those sections of Title 35, U.S. Code not included in this action can be found in a prior Office action.
- Claims 1-9 and (newly added) 11-14 are rejected under 35 U.S.C. 103(a) as being unpatentable over Schlumpf (Canadian Patent 2016447) in view of Shaw et al (USP 4643940), Hosoda et al (USP 3812225) and Figini et al (USP 4680318).
- This rejection (also to be applied to new claims 11-14) is incorporated here by reference from section 4, pages 3-5, of prior office, paper no. 5).
- Applicant's arguments filed May 11, 2000 (paper no. 9) have been fully considered but they are not persuasive.

(I) 17 fig 2 cr 2

[0003] In order to ensure adequate penetration of the impregnation liquors into the paper or cardboard, the resin solutions must be processed from aqueous or alcohol dilution. Owing to the high level of cellulose swelling in aqueous systems, the foils and edgings produced from high aqueous dilution are brittle, exhibit a high level of water absorption, and even in the coated state have a surface whose visual appeal is low. The procedure described in DE-A 23 09 334, comprising impregnating liquors diluted with C.sub.1 to C.sub.4 alcohols, does give foils and edgings having good performance properties but requires complex measures for reprocessing the waste gas. The route to a solution that is described in DE-A 44 39 156, modifying melamine resins with guanamines, makes it possible to carry out impregnation from purely aqueous impregnating liquors. A disadvantage, however, is the significantly higher cost of the amino resin, resulting from the use of the guanamines.

[0004] The formaldehyde emissions of finished foils and edgings after manufacturing are determined following storage under standard climatic conditions (23.degree. C., 50% relative atmospheric humidity) in accordance with the standard EN 717-2 (FESYP method, gas analysis). Foils and edgings with values of less than 3.6 mg/(h.multidot.m.sup.2) by the FESYP method meet the limit of the standard ("E 1"). The rates of emission found remain more or less constant even after several weeks of storage under standard climatic conditions. Formaldehyde emissions observed on the films and edgings arise due to the use of urea-formaldehyde and/or melamine-formaldehyde resins in the impregnating liquors for impregnating the paper or cardboard sheets and/or in the coating formulations for coating the films and edgings. By using particularly low-formaldehyde urea-formaldehyde and/or melamine-formaldehyde resins (with low formaldehyde clearance) it is possible to reduce the formaldehyde emissions as measured by the FESYP method (standard climatic conditions) to values around 2 mg/(h.multidot.m.sup.2).

[0005] It has surprisingly now been found that when edgings produced in accordance with the prior art and originally (before the commencement of storage) satisfying the classification E1 ("E1 edgings"), with initial formaldehyde emission values of from 1.0 to 3.5 mg/(h.multidot.m.sup.2), are stored under nonstandardized climatic conditions, at customary summer temperatures and atmospheric humidities, the formaldehyde emissions rise in the course of a few weeks to values of in some cases much higher than 3.5 mg/(h.multidot.m.sup.2), and so the edgings no longer meet the E1 criterion. This unexpectedly high increase in the formaldehyde emissions was confirmed by storage under defined conditions in a tropical climate (35.degree. C., 90% relative atmospheric humidity), with measurement being carried out only after 3-day reconditioning under standard climatic conditions following storage under the tropical climatic conditions.

[0006] From the prior art it is known that the amount of free formaldehyde and also the formaldehyde emissions may be reduced by adding formaldehyde scavengers such as urea and urea derivatives, for example. For instance, according to DE-A 38 37 965, finished foils and edgings with formaldehyde emissions that are negligible as determined in accordance with DIN 52368 may be produced by adding urea to the melamine-formaldehyde condensation product. Regarding the behavior during and after storage under tropical climatic conditions, however, no information is given. According to DE-A 34 03 136, mixtures of organic hydroxy compounds and an amide are suitable for use as formaldehyde-binding agents in boards made from wood cellulose materials. The use of these mixtures as formaldehyde scavengers in finished foils and edgings is not described. The addition of the mixtures described in DE-A 34 03 136 to amino resins that are used to produce finished foils and edgings leads to a marked deterioration in the flexibility of the finished foils and edgings produced with them. The use of formaldehyde scavengers known from the literature, such as urea, ethyleneurea and propyleneurea, resulted in finished foils and edgings which met the E1 criterion under standard climatic conditions but which markedly exceed the E1 limit of 3.5 mg/(h.multidot.m.sup.2) under tropical climatic conditions.

[0007] It is therefore an object of the present invention to develop amino-resin mixtures for producing films and edgings, which can be used to produce, relative to the prior art, finished foils and furniture edgings with significantly reduced formaldehyde emission when stored under tropical climatic conditions, while retaining the required performance properties.

SUMMARY OF THE INVENTION

Art Unit: 1711

Applicant's arguments do not comply with 37 CFR 1.111(c) because they do not clearly point out the patentable novelty which he or she thinks the claims present in view of the state of the art disclosed by the references cited or the objections made. Further, they do not show how the amendments avoid such references or objections.

On page 6, paragraph 2 of above response paper no. 9, the applicants argue that Schlumpf teaches away from claimed invention because Schlumpf discloses solids at least 92.1%. While there is a difference of only 0.1% (between claimed 92.0% and disclosed 92.1%), this difference fails to distinguish patentably the instant invention from the prior art, particularly because the applicants have not established the criticality of the upper limit viz. 92.0%.

Applicants' further argue in the next paragraph on the same page, that Schlumpf does not teach claimed particle size distribution this argument is not persuasive. On page 9, Schlumpf discloses a calcium carbonate of a mean statistical particle diameter of 0.5 - 50 microns. This range overlaps that of calcium carbonate (viz. 0.3 to 18 microns) in example 1 in instant specification page 14. Furthermore, also the applicants have not shown any criticality of having the only the claimed particle size distribution.

On page 6, paragraph 4 of same paper, the applicants state that apparatus of Schlumpf cannot degas the composition and therefore Schlumpf teaches away from inclusion of a blowing agent. Whether the apparatus can or cannot degas the composition has no influence at all.

[0014] Likewise suitable as formaldehyde-binding component B2 are reaction products containing urethane groups, said products being obtained by reacting polyhydroxyl compounds B21, including the compounds mentioned under B12 and also aliphatic polyhydroxy amines B211 having from 2 to 6 hydroxyl groups and 1 to 4 nitrogen atoms, attached in an aminelike manner, per molecule and containing no free amine-type hydrogen atoms, such as N-methyldiethanolamine, N,N,N',N'-tetrakis(2-hydroxyethyl)ethylenediamine and triethanolamine with monofunctional or polyfunctional aliphatic, cycloaliphatic or aromatic isocyanates B22, such as hexamethylene diisocyanate, for example.

[0015] The addition of formaldehyde-binding auxiliary B (calculated by mass without solvents or diluents, i.e., on a 100% basis) amounts to from 2 to 50 parts by weight per 100 parts by weight of amino resin (likewise on a 100% basis). In addition to the reduction in formaldehyde, it is also found when using a reaction product of hydroxy amines and diisocyanate, such as the reaction product of triethanolamine with hexamethylene diisocyanate in Example 11, that the pot life is extended significantly.

[0016] The acrylic resin dispersion C is a dispersion of an acrylic copolymer in water, preparable for example by emulsion copolymerization of olefinically unsaturated monomers, the monomer mixture used for its preparation comprising a predominant fraction (more than 50% of its mass) of what are known as acrylic monomers, i.e., acrylic or methacrylic acid and derivatives thereof, especially esters with aliphatic alcohols having 1 to 10 carbon atoms, esters with aliphatic polyhydroxy compounds having 2 to 10 carbon atoms and at least two hydroxyl groups per molecule, and the nitriles of said acids. Preferred acrylic monomers among the esters are methyl, ethyl, n-butyl, t-butyl, hexyl and 2-ethylhexyl (meth)acrylate, hydroxyethyl and hydroxypropyl (meth)acrylate. It is additionally possible for copolymerizable monomers such as styrene and other aromatic vinyl compounds, esters or monoesters of olefinically unsaturated dicarboxylic acids such as, in particular, maleic acid, vinyl esters such as vinyl acetate or vinyl Versatate, vinyl halides or vinyl ethers to be copolymerized. The synthetic resin dispersions usually have mass fractions of solids of from 25 to 85%; they are added to the amino resin or else to the impregnating liquor itself. The ratio of the mass of the acrylic copolymer in the acrylic dispersion to the mass of the amino resin in its aqueous solution or dispersion may within the composition be from 0 to 150:100, preferably from 20 to 140:100.

[0017] The amino resin compositions are used to impregnate absorbent papers or cardboards. The amount of amino resin composition introduced is usually such that the mass per unit area of the paper or cardboard following impregnation and subsequent drying increases by a factor of from 1.3 to 2.5, preferably from 1.4 to 1.8.

[0018] The use of the mixtures of the invention leads to a significant reduction in the formaldehyde emissions from the foils and edgings stored under tropical climatic conditions.

#### EXAMPLES

[0019] Preparation of a Partially Etherified Melamine-formaldehyde (MF) Resin

[0020] A 30 l laboratory vessel with stirrer, reflux condenser and thermometer was charged with 6717 g (87.2 mol) of 39% strength aqueous formaldehyde and this initial charge was heated to 68.degree. C. Then 31.5 ml of 2 N sodium hydroxide solution were added followed immediately by 3450 g (27.4 mol) of melamine. Because of the exothermic reaction of melamine and formaldehyde, the mixture rose in temperature to about 83.degree. C. and was held at this temperature until all of the melamine had dissolved. It was then cooled to 55.degree. C. and 16560 g (517 mol) of methanol and 30 ml of 53% strength nitric acid were added. The reaction mixture was heated to 59.degree. C. and stirred at this temperature until a clear solution was formed. After a further 30 minutes, the reaction was terminated by adding about 140 ml of 2 N NaOH. The pH was adjusted to 10. Excess methanol was removed by distillation under reduced pressure (generated by a water jet pump) and the mass fraction of solids of the resin was adjusted to 75% (measured on a 2 g sample, dried at 120.degree. C. for 1 h in a glass dish). The resin had the following characteristics: content (mass fraction of solids): 75%; viscosity at 23.degree. C.: 480 mPa.multidot.s, mass fraction of free formaldehyde: 0.17%;

1. The request filed on May 11, 2000 (paper no. 15) for a Continued Prosecution Application (CPA) under 37 CFR 1.53(d) based on parent Application No. 09/047161 is acceptable and a CPA has been established. An action on the CPA follows.

Claims 1-16 are pending, out of which claim 15 & 16 are non-elected, leaving 1-14 to be examined.

2. Rejection of claim 1 under 35 U.S.C. 102(b) set forth in section 3, pages 3 & 4 of office action of January 25, 1999 (paper no. 5) and maintained in later office actions, is now withdrawn as a result of amendment. That of claim 13 is however maintained.

3. The text of those sections of Title 35, U.S. Code not included in this action can be found in a prior Office action.

Claims 1-14 are rejected under 35 U.S.C. 103(a) as being unpatentable over Taubitz et al (USP 4774286) in view of Levine et al (USP 4321087), Kohara et al (USP 5302656) and Clark et al (USP 5510398).

(All references are of record in prior office actions).

Taubitz discloses thermoplastic molding materials based on polycarbonates, polyesters and a graft copolymer (abstract). The graft copolymer is based on acrylic acid esters, methacrylic acid esters and/or conjugated dienes and an epoxy-containing monomer (col. 3, lines 1-12). Elastomeric constituents viz rubbers can be included also (col. 4, lines 3-13). (These rubbers read on those of instant claim 4). Metal flakes and metal fibers and mineral particles are also added to such molding material (col. 4, lines 44-50 and 60-63).

[0008] The invention provides aqueous amino resin compositions comprising amino resins A, formaldehyde-binding additives (auxiliaries) B, which may comprise hydroxyl group-containing polyurethanes B13, and, if desired, acrylic resins C in the form of aqueous dispersions, and water.

#### DETAILED DESCRIPTION OF THE INVENTION

[0009] Where the component B consists only of at least one organic hydroxyl compound which is soluble in water or a monohydric alcohol having 1 to 4 carbon atoms and is selected from dihydric, trihydric and pentahydric alcohols containing up to 6 carbon atoms, pentaerythritol and sorbitol, monosaccharides containing up to 6 carbon atoms, disaccharides containing up to 12 carbon atoms, polysaccharides having an Ostwald viscosity of up to 200 mPa.multidot.s at 25.degree. C. and a concentration corresponding to 37% refraction, monohydric and polyhydric aromatic alcohols containing only one benzene ring, and monohydric and polyhydric phenols, and of at least one amide which is soluble in water or a monohydric alcohol having 1 to 4 carbon atoms and is selected from aliphatic amides containing up to 6 carbon atoms and aromatic amides containing only one benzene ring, the presence of at least one of the components B13 and C in the composition is mandatory.

[0010] The amino resins A are water-soluble melamine resins, urea resins or mixed melamine-urea condensates which are etherified with C.sub.1 to C.sub.4 alcohols. In the compositions of the invention it is also possible to use those melamine resins in which a fraction (up to 20% of its mass) of the melamine has been replaced by other triazines such as acetoguanamine, caprino-guanamine or benzoguanamine. Preferred resins, however, contain less than 10%, in particular less than 5%, of other triazines, measured on the same scale. Particular preference is given to straight melamine resins or to their cocondensates with urea. The resins are etherified at least partially with the abovementioned alcohols, especially methanol, n-butanol and isobutanol. Particular preference is given to methanol-etherified amino resins.

[0011] Particular preference is given above all to melamine resins having an amount-of-substance ratio (molar ratio) of melamine to urea to formaldehyde to methanol of 1 mol: (0 to 2 mol):(0.8 to 5.8 mol):(0.8 to 5.5 mol).

[0012] The preparation of the amino resins A is widely known. First of all, methylation and condensation are carried out by adding formaldehyde to the amino resin formers at pH values from 7 to 10 and temperatures from 40 to 110.degree. C., after which the etherifying alcohol is added and reaction is continued at pH values from 1 to 7 and temperatures from 30 to 80.degree. C. The condensation conditions and etherification conditions are guided by the water dilutability desired for the resin, which amounts to at least 1 part by weight of resin to 5 parts by weight of deionized water, and by the required penetration properties.

[0013] As component B, formaldehyde-binding auxiliaries are added. These auxiliaries are selected from mixtures B1 of organic amides B11 having up to 10 carbon atoms and from one to four nitrogen atoms attached in amidelike or imidelike manner, such as urea and/or urea derivatives such as thiourea, ethyleneurea (2-imidazolidinone), propyleneurea, acetyleneurea (glycoluril), and also formamide, acetamide, benzamide, oxalamide, succinimide, malonamide and dicyandiamide, and polyhydroxyl compounds B12 selected from aliphatic linear and branched compounds B121 having from 2 to 6 hydroxyl groups and 1 to 10 carbon atoms, such as glycol, 1,2- and 1,3-propylene glycol, neopentyl glycol, glycerol, trimethylolpropane, ditrimethylolpropane, erythritol, pentaerythritol, dipentaerythritol, sorbitol and mannitol, monosaccharides B122 having up to 6 carbon atoms, and disaccharides B123 having up to 12 carbon atoms, and, if desired, water-soluble or water-dispersible, hydroxyl-containing urethane compounds B13. These urethane compounds are of low mol mass (number-average molar mass M.sub.n from 150 to 5000 g/mol, preferably from 300 to 4000 g/mol) and contain hydrophilic groups which are preferably nonionic, especially building blocks derived from glycol or from oligoethylene or polyethylene glycol. Examples of suitable compounds are adducts of aliphatic linear or cyclic diisocyanates, such as 1,2-diisocyanatoethane and 1,6-diisocyanatohexane, with ethylene glycol, diethylene glycol or mixtures thereof with 1,2- or 1,3-propylene glycol, these latter hydrophobic diols being used only in fractions (e.g., less than 25% of the mass of diols overall) such that the adduct remains soluble or dispersible, respectively, in water.

Art Unit: 1711

Any inquiry of a general nature or relating to the status of this application or proceeding should be directed to the receptionist whose telephone number is (703) 308-0661.

U.K. Rajguru/om  
August 14, 2000



water dilutability: unlimited.

[0021] Auxiliary BA: Mixture of Glycerol and Urea

[0022] 100 g of urea were introduced with stirring into 100 g of glycerol and the mixture was heated to 90.degree. C. After the urea had dissolved, it was cooled to 20.degree. C. This gave a mixture with a pastelike consistency.

[0023] Auxiliary BB: Mixture of Glycerol, Urea and Polyurethanediol

[0024] 100 g of urea and 67 g of 88% strength solution of urethanediol (number-average molar mass  $M_{sub.n}$  320 g/mol, OH number 350 mg/g, urethane group content 37 cg/g) were introduced with stirring into 100 g of glycerol and the mixture was heated to 90.degree. C. Following dissolution, the mixture was cooled to 20.degree. C. This gave a mixture with a pastelike consistency.

[0025] Auxiliary BC: Reaction Product of Trimethylolpropane (TMP) and Hexamethylene Diisocyanate (HDI)

[0026] In a suitable reaction vessel with water separator, 100 g of TMP were melted, after which nitrogen was passed over the material at 140.degree. C. with stirring for 3 hours in order to eliminate traces of water. The system was then cooled to 60.degree. C. and 8 g of HDI was added slowly dropwise with vigorous stirring. The temperature was maintained until the mass fraction of isocyanate groups in the reaction product (NCO value) had fallen below 0.1%. Then the mass fraction of solids was adjusted to 85% using water. The reaction mixture was a colorless solution of low viscosity.

[0027] Auxiliary BD: Reaction Product of Tripropylene Glycol and Hexamethylene Diisocyanate

[0028] In a suitable reaction vessel with water separator, 100 g of tripropylene glycol were introduced, after which nitrogen was passed over the material at 140.degree. C. with stirring for 3 hours in order to eliminate traces of water. The system was then cooled to 40.degree. C. and 5 g of HDI were added slowly dropwise with vigorous stirring. The temperature was maintained until the NCO value had fallen below 0.1%. Then the mass fraction of solids was adjusted to 85% using water. The reaction mixture was a pale yellow solution of low viscosity.

[0029] Auxiliary BE: Reaction Product of Glycerol and Hexamethylene Diisocyanate

[0030] In a suitable reaction vessel with water separator, 100 g of glycerol were introduced, after which the water was removed azeotropically at 140.degree. C. for 5 hours using special boiling-point spirit 80/120 as azeotrope former. Following the removal of the azeotrope former (by distillation), the system was then cooled to 60.degree. C. and 5 g of HDI were added slowly dropwise with vigorous stirring. The temperature was maintained until the NCO value had fallen below 0.1%. The reaction mixture was a yellow solution of medium viscosity.

[0031] Auxiliary BF: Reaction Product of Triethanolamine and Hexamethylene Diisocyanate

[0032] In a suitable reaction vessel with water separator, 100 g of triethanolamine were introduced, after which the water was removed azeotropically at 140.degree. C. for 5 hours using special boiling-point spirit 80/120 as azeotrope former. Following the removal of the azeotrope former, the system was then cooled to 40.degree. C. and 15 g of HDI were added slowly dropwise with vigorous stirring. The temperature was maintained until the NCO value had fallen below 0.1%. The reaction mixture was a yellow solution of medium viscosity.

[0033] Performance Testing:

[0034] The MF resin described above was used in each of the examples. The acrylic dispersion used was a dispersion based on a copolymer of methyl methacrylate, butyl acrylate, hydroxyethyl methacrylate, acrylic acid and styrene, having a hydroxyl number of about 120 mg/g and a mass fraction of solids of about 50%, which was diluted if necessary to the lower specified value (45%).

Inventive Examples 1-4 and Comparative Examples 1-4

Art Unit: 1711

Tanbitz does not mentioned the (claimed) particle size of metallic particles and mineral particles, average diameter and average aspect ratio of fibers.

Levine discloses that a preferred metallic pigment should have a diameter of 10-20

microns (see Levine, col. 5, lines 30-34).

Kohara discloses that the average diameter of a fibrous filler is 0.1 to 100 micrometers,

and the length is from 5 micrometers to 20 millimeters. Average article diameter of powdery

filler is 0.01 micrometer to 1 millimeter (see Kohara, col. 4, lines 27-38).

Clark discloses thermoplastic compositions having a speckled surface appearance; This

appearance results from the use of nondispersing pigments (abstract; col. 10, lines 1-2). It is

preferred that these pigments possess a large aspect ratio greater than about 20 (col. 10, lines 33-

38).

Based on the teachings of Levine, Kohara and Clark, it is the examiner's position that it

would have been obvious to one ordinary skill in the art to use in the composition of Tanbitz

metallic particles of claimed particle size, fibers of claimed average diameter and average aspect

ratio and interala particles of claimed average particle size in order to produce products having

bright reflective appearance, enhanced hiding power, improved crack resistance and moldability

as well as better speckled appearance.

4. Any inquiry concerning this communication or earlier communications from the

examiner should be directed to U.K. Rajguru whose telephone number is (703) 308-3224. The

examiner can normally be reached on Monday - Friday from 9:30 am to 6:00 pm.

[0036] To determine the formaldehyde emissions, the edging samples were conditioned for 3 days under standard climatic conditions (23.degree. C., 50% relative atmospheric humidity) prior to each measurement. To determine the formaldehyde emissions under tropical climatic conditions, the edgings were stored for 1 to 4 weeks at 35.degree. C. and 90% relative atmospheric humidity, after which they were reconditioned for 3 days under standard climatic conditions, prior to the actual measurement. The formaldehyde emissions were measured in accordance with EN 717-2. Table 1 lists the resulting formaldehyde emissions in mg/(h.multidot.m.sup.2) as averages of the 1- to 4-hour values:

[0037] The edgings produced in accordance with Inventive Examples 2 and 4 meet the E1 criterion (i.e., 3.6 mg/h/m.sup.2) even after one week of storage under tropical climate conditions.

[0038] 1.5 g of p-toluenesulfonic acid were added to 100 g of the above-described MF resin, along with the auxiliaries indicated in Table 2 for the individual application examples. These undiluted liquors were used to impregnate papers (typical edgebanding cardboard; 200 g/m.sup.2) by means of knife application from the decorative side, and the impregnated edgings were dried at 160.degree. C. to a residual moisture content of about 1.5%. Thereafter, the basis weight was about 305 g/m.sup.2. The impregnated edgings thus obtained were coated with an aqueous acid-curing varnish (see above) and dried at a temperature of 160.degree. C. to a residual moisture content of 1.5%. The varnish add-on was about 20 g/m.sup.2. With regard to their performance properties, the edgings obtained in each case met the requirements.

2 TABLE 2 Directly after After After After After Auxiliary prep. 1 wk 2 wks 4 wks  
6 wks Inventive 12 g BB 1.0 2.2 2.1 1.8 1.8 Example 5 Comp. Ex. 5 -- 1.0 3.3 2.4  
2.9 2.9

[0040] 200 g of 50% acrylic dispersion and 1.7 g of p-toluenesulfonic acid were added to 100 g of the above-described MF resin, along with the amounts of auxiliaries indicated in Table 3 for the individual application examples and also PEG 400 (polyethylene glycol having a number-average molar mass  $M_{sub.n}$  of about 400 g/mol). Following dilution to a liquor concentration of 47% (mass fraction of the resins in the aqueous liquor), these resins were used to impregnate papers (typical edgebanding cardboard; mass per unit area about 200 g/m<sup>sup.2</sup>) and the impregnated edgings were dried at 170.degree. C. to a residual moisture content of about 1.6%. Following impregnation, the final weight was about 330 g/m<sup>sup.2</sup>. The

Art Unit 1711

If attempts to reach the examiner by telephone are unsuccessful, the examiner's

supervisor, Jim Seidleck, can be reached on (703) 308-2462. The fax phone number for the

organization where this application or proceeding is assigned is (703) 305-3599.

Any inquiry of a general nature or relating to the status of this application or proceeding

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U.K. Rajguru/om  
August 14, 2000

conditioned for 3 days under standard climatic conditions (23.degree. C., 50% relative atmospheric humidity) prior to each measurement. To determine the formaldehyde emissions under tropical climatic conditions, the edgings were stored for 1 week at 35.degree. C. and 90% relative atmospheric humidity, after which they were reconditioned for 3 days under standard climatic conditions, prior to the actual measurement. The formaldehyde emissions were measured in accordance with EN 717-2. Table 5 lists the resulting formaldehyde emissions as averages of the 1- to 4-hour values. The flexibility of the edgings was assessed at room temperature with the aid of the flexural test. The parameter reported was the band radius at which the edging still just did not fracture. As is evident from Table 5, the formaldehyde emissions can be reduced significantly relative to Comparative Example 10 while retaining a very low band radius. The desired formaldehyde reduction cannot be achieved by adding small amounts of urea and sorbitol (Comparative Example 11). The pot life is the time taken for the impregnating liquor, stored at 30.degree. C., to obtain a viscosity (measured as the efflux time in accordance with DIN 53211 at 23.degree. C.) of more than 60 seconds or for the penetration time with a defined test paper to rise to more than 70 seconds. The auxiliary BF has a considerable advantage as compared with the prior art, with regard to formaldehyde emissions and pot life.

TABLE 5 Pot Directly life at Band after After 30.degree. C. radius Auxiliary 100% PEG 400 Sorbitol Urea prep. 1 wk in h in mm Inventive 9g BE 27 g 1.1 g 1.1 g 0.46 1.47 8 6 to Example 10 8 Inventive 9g BF 27 g 1.1 g 1.1 g 0.17 0.46 >30 5 to Example 11 6 Comp. Ex. 10 -- 36 g -- -- 0.75 1.98 6 5 to 6 Comp. Ex. 11 -- 36 g 1.1 g 1.1 g 0.58 1.82 7 5 to 6

#### CLAIMS:

1. An aqueous amino resin composition comprising amino resins A, formaldehyde-binding additives (auxiliaries) B, which if desired comprise hydroxyl-containing polyurethanes B13, if desired, acrylic resins C in the form of aqueous dispersions, and water, with the proviso that the presence of at least one of the components B13 and C in the composition is mandatory where the component B consists only of at least one organic hydroxyl compound which is soluble in water or a monohydric alcohol having 1 to 4 carbon atoms and is selected from dihydric, trihydric and pentahydric alcohols containing up to 6 carbon atoms, pentaerythritol and sorbitol, monosaccharides containing up to 6 carbon atoms, disaccharides containing up to 12 carbon atoms, polysaccharides having an Ostwald viscosity of up to 200 mPa.multidot.s at 25.degree. C. and a concentration corresponding to 37% refraction, monohydric and polyhydric aromatic alcohols containing only one benzene ring, and monohydric and polyhydric phenols, and of at least one amide which is soluble in water where a monohydric alcohol having 1 to 4 carbon atoms and is selected from aliphatic amides containing up to 6 carbon atoms and aromatic amides containing only one benzene ring.
2. The aqueous amino resin composition as claimed in claim 1, wherein the amino resins A are water-soluble melamine resins, urea resins or melamine-urea cocondensates which are etherified with C.sub.1 to C.sub.4 alcohols.
3. The aqueous amino resin composition as claimed in claim 1, wherein the amino resins A are methanol-etherified melamine resins or melamine/urea resins having an amount-of-substance ratio (molar ratio) of melamine to urea to formaldehyde to methanol of 1 mol:(0 to 2 mol):(1.8 to 5.8 mol):(0.8 to 5.5 mol).
4. The aqueous amino resin composition as claimed in claim 1, wherein the ratio of the mass of component B to the mass of the amino resin A in the mixture is from 1 to 30:100.
5. The aqueous amino resin composition as claimed in claim 1, wherein the formaldehyde-binding auxiliaries B are selected from mixtures B1 of organic amides B11 having up to 10 carbon atoms and from one to four nitrogen atoms, attached in amidelike or imidelike manner, and polyhydroxyl compounds B12 selected from aliphatic linear and branched compounds B121 having from 2 to 6 hydroxyl groups and 1 to 10 carbon atoms, monosaccharides B122 having up to 6 carbon atoms, and disaccharides B123 having up to 12 carbon atoms, and, if desired, water soluble or water dispersible, hydroxyl-containing urethane compounds B13.
6. The aqueous amino resin composition as claimed in claim 1 or 5, wherein urethane compounds B13 are of low molecular mass, having an above average molar



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mass M.sub.n of from 150 to 5000 g/mol, and contain hydrophilic groups.

7. The aqueous amino resin composition as claimed in claim 1, wherein formaldehyde-binding auxiliaries B used comprise the components B2, which are obtainable by reacting polyhydroxyl compounds B21 selected from aliphatic linear and branched compounds B121 having from 2 to 6 hydroxyl groups and 1 to 10 carbon atoms, monosaccharides B122 having up to 6 carbon atoms, and disaccharides B123 having up to 12 carbon atoms, and also aliphatic aliphatic polyhydroxy amines B211 having from 2 to 6 hydroxyl groups and 1 to 4 nitrogen atoms, attached in an aminelike manner, per molecule, and containing no free amine-type hydrogen atoms, with monofunctional or polyfunctional aliphatic, cycloaliphatic or aromatic isocyanates B22.

8. The aqueous amino resin composition as claimed in claim 1, wherein the acrylic resins C are aqueous dispersions of an acrylic copolymer in water, the parent monomer mixture containing more than 50% of its mass of acrylic monomers selected from acrylic acid and methacrylic acid, their esters with aliphatic alcohols having 1 to 10 carbon atoms, their esters with aliphatic polyhydroxy compounds having 2 to 10 carbon atoms and at least two hydroxyl groups per molecule, and the nitrites of said acids.

9. The aqueous amino resin composition as claimed in claim 8, wherein the ratio of the mass of the acrylic copolymer to the mass of the amino resin in the mixture is from 0 to 150:100.

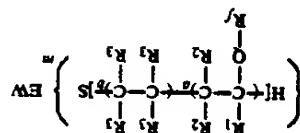
10. The use of the aqueous amino resin composition as claimed in claim 1 for impregnating paper or cardboard for use as finished foils or edgings.

11. A finished foil or edging comprising cardboard or paper impregnated with the aqueous amino resin composition as claimed in claim 1.

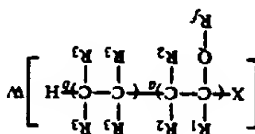
12. The finished foil or edging as claimed in claim 11, wherein the mass per unit area of the paper or cardboard following impregnation and subsequent drying is greater by a factor of from 1.3 to 2.5 than that of the substrate that is used.

-continued

wherein  $q$  is an integer from 1 to about 10;  $R_1$  is an  $(n+q)$ -valent organic group such as 2,4-10-ylene; and  $n$ ,  $m$ ,  $a$ ,  $b$ ,  $E$ ,  $R_1$ ,  $R_2$ ,  $R_3$ ,  $X$ , and  $Z$  are as defined above for Formulas I-III. In a compound of Formulas Ia or IIa, optionally at least one of  $R_1$ ,  $R_2$ ,  $R_3$ ,  $Q$ , and  $X$  comprises a group that can impart soft hand, stain repellency, or a durable property when the compound is applied to a fibrous substrate. This invention also provides a method of stabilizing an aqueous liquid dispersion, comprising the step of including in the dispersion a fluorochemical emulsifier of Formula IV or IVA.



IV



IVA

wherein  $R_1$ ,  $R_2$ ,  $R_3$ ,  $Q$ ,  $R_4$ ,  $R_5$ ,  $E$ ,  $X$ ,  $a$ ,  $b$ , and  $m$  are as defined above in connection with Formula III (with the obvious proviso that all bonds defined as bonds to  $Z$  in Formula III are bonds to  $W$  in Formulas IV and IVA), and wherein  $W$  represents the hydrophilic portion of

- 1) an anionic surfactant;
- 2) a cationic surfactant;
- 3) a non-ionic surfactant; or
- 4) an amphoteric surfactant.

in an amount effective to stabilize the dispersion. These emulsifiers find particular use in the aqueous dispersions of the invention.

As described above and further illustrated in Formulas I-III, a fluorochemical composition of the invention comprises a fluorinated compound that generally has four principal portions: a fluorochemical oligomeric portion, an organic moiety, and a group that can impart soft hand, stain repellency, or a durable property when the compound is applied to a fibrous substrate. The fluorochemical oligomeric portion and the organic moiety are linked together by the isocyanate-derived linking group. The isocyanate-derived linking group can result from the reaction of a nucleophilic, such as an alcohol, an amine, or a thiol, with an isocyanate or with an isocyanate dimer, trimer, oligomer, or the like. Preferred among such linking groups are ureylene, urethanylene, allophanylene, biuretylene, guanidinylene, oxazolidinonylene, acylamino, and sulfur analogs thereof.

A salient component of the fluorochemical oligomeric portion is the fluorophilic group, designated herein as  $R_f$ . A fluorinated compound contains a plurality of  $R_f$  groups (e.g., from 2 to about 50) proximal to one another and preferably contains from about 5 per-

cent to about 80 percent, more preferably from about 20 percent to about 55 percent fluorine by weight based on the total weight of the compound, the loci of the fluorine being essentially in the  $R_f$  groups.  $R_f$  is a stable, inert, non-polar, preferably saturated, monovalent moiety which is both oleophobic and hydrophobic.  $R_f$  preferably contains at least about 3 carbon atoms, more preferably about 6 to about 14 carbon atoms.  $R_f$  can contain straight chain, branched chain, or cyclic fluorinated alkylene groups or combinations thereof or combinations thereof with straight chain, branched chain, or cyclic alkylene groups.  $R_f$  is preferably free of polymerizable olefinic unsaturation and can optionally contain catenary heteroatoms such as oxygen, divalent or hexavalent sulfur, or nitrogen. It is preferred that  $R_f$  contain about 40% to about 78% fluorine by weight, more preferably about 50% to about 78% fluorine by weight. The terminal portion of the  $R_f$  group contains a fully fluorinated terminal group. This terminal group preferably contains at least 7 fluorine atoms, e.g.,  $CF_3CF_2CF_2-$ ,  $(CF_2)_2CF-$ ,  $(CF_2)_3CF-$ , or the like. Perfluorinated alkyl groups (i.e., those of the formula  $C_nF_{2n+1}$ ) are the most preferred embodiments of  $R_f$ .

The aliphatic backbone of the fluorochemical oligomeric portion comprises a sufficient number of polymerized units to render the portion oligomeric. The aliphatic backbone preferably comprises from 2 to about 20 polymerized units derived from fluorinated monomers (i.e., monomers containing a fluorinated organic group  $R_f$  as defined above), and from 0 to about 20 polymerized units derived from non-fluorinated monomers. In instances where only units derived from fluorochemical monomers are present, it is preferred that the aliphatic backbone comprise from 2 to about 8, most preferably about 4, polymerized units. In instances where both fluorochemical and non-fluorinated monomers are used, likewise the aliphatic backbone preferably comprises from 2 to about 8, most preferably about 4, polymerized units. The ratio of moles of fluorochemical monomer to moles of polymerized units derived from fluorochemical monomer to moles of polymerized units derived from non-fluorinated monomer is preferred to be from 20:1 to about 1:4, preferably from about 10:1 to about 2:3, and most preferably about 4:1.

The fluorochemical oligomeric portion is linked to the organic portion by a linking group designated as  $Z$  in the formulas used herein.  $Z$  is a non-polymeric isocyanate-derived linking group that can result from the reaction of a nucleophile such as an alcohol, an amine, or a thiol with an isocyanate or an isocyanate dimer, trimer, oligomer, or the like. Suitable isocyanate-



impregnated edgings obtained in this way were coated with an aqueous acid curing varnish (see above) and dried at a temperature of 160.degree. C. for 60 seconds. The varnish add-on was about 16 g/m.sup.2. With regard to their performance properties, each of the edgings obtained met the requirements.

[0041] To determine the formaldehyde emissions, the edging samples were conditioned for 3 days under standard climatic conditions (23.degree. C., 50% relative atmospheric humidity) prior to each measurement. To determine the formaldehyde emissions under tropical climatic conditions, the edgings were stored for 1 week at 35.degree. C. and 90% relative atmospheric humidity, after which they were reconditioned for 3 days under standard climatic conditions, prior to the actual measurement. The formaldehyde emissions were measured in accordance with EN 717-2. Table 3 lists the resulting formaldehyde emissions in mg/(h.multidot.m.sup.2) as averages of the 1- to 4-hour values:

3 TABLE 3 Auxiliary as 100% Directly After substance PEG 400 after prep. 1 week  
Inventive 20 g BC 20 g 0.97 2.61 Example 6 Inventive 20 g BD 20 g 1.28 2.63  
Example 7 Comp. Ex. 6 -- 40 g 1.52 3.49 Comp. Ex. 7 20 g polypropylene 20 g 1.82  
2.93 glycol (M.sub.n < 400 g/mol)

Inventive Examples 8 and 9 and Comparative Examples 8 and 9

[0042] 1.6 g of 45% acrylic dispersion and 1.0 g of p-toluenesulfonic acid were added to 100 g of the above-described MF resin, along with the auxiliaries indicated in Table 4 for the individual application examples. Following dilution to a liquor concentration of 75%, these liquors were used to impregnate papers (typical edgebanding cardboard; 200 g/m.sup.2) and the impregnated edgings were dried at 180.degree. C. for 90 seconds. The final weight was about 335 g/m.sup.2. With regard to their performance properties, the uncoated edgings obtained met the requirements.

[0043] To determine the formaldehyde emissions, the edging samples were conditioned for 3 days under standard climatic conditions (23.degree. C., 50% relative atmospheric humidity) prior to each measurement. To determine the formaldehyde emissions under tropical climatic conditions, the edgings were stored for 1 week at 35.degree. C. and 90% relative atmospheric humidity, after which they are reconditioned for 3 days under standard climatic conditions, prior to the actual measurement. The formaldehyde emissions were measured in accordance with EN 717-2. Table 4 lists the resulting formaldehyde emissions as averages of the 1- to 4-hour values. The flexibility of the edgings was assessed at room temperature with the aid of the flexural test. The parameter reported was the band radius at which the edging still just did not fracture. As is evident from Table 4, the formaldehyde emissions can be reduced significantly relative to Comparative Example 8 while retaining a very low band radius. The desired formaldehyde reduction cannot be achieved by adding small amounts of urea and sorbitol. Added at higher levels (Comparative Example 9a), there is a deterioration in the flexibility of the edging (larger band radius).

[0044] The table indicates the formaldehyde emission in mg/(h.multidot.m.sup.2) and the band radius in mm.

4 TABLE 4 Auxiliary Directly Band as 100% PEG after After radi- substance 400  
Sorbitol Urea prep. 1 wk us Inventive 9 g BC 27 g -- -- 0.64 1.57 5 Example 8  
Inventive 9 g BC 27 g 1.1 g 1.1 g 0.49 1.51 5 Example 9 Comp. Ex. 8 -- 36 g -- --  
0.97 2.51 6 Comp. Ex. 9 -- 36 g 1.1 g 1.1 g 0.80 2.22 5 Comp. Ex. -- 36 g 3.8 g  
3.8 g -- -- 8 9a

Inventive Examples 10 and 11 and Comparative Examples 10 and 11

[0045] 1.7 g of 45% acrylic dispersion and 1.3 g of p-toluenesulfonic acid were added to 100 g of the above-described MF resin, along with the auxiliaries indicated in Table 5 for the individual application examples. Following dilution to a liquor concentration of 75%, these liquors were used to impregnate papers (typical edgebanding cardboard; 200 g/m.sup.2) and the impregnated edgings were dried at 180.degree. C. for 90 seconds. The final weight was about 335 g/m.sup.2. With regard to their performance properties, the uncoated edgings obtained met the requirements.

[0046] To determine the formaldehyde emissions, the edging samples were



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Jan 17, 1989

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TITLE: Bis-(2,2,6,6-tetramethyl-piperidyl) diimides as light stabilizers for synthetic polymers

BSPR:

or Y and R.sub.7 together with the N-atom to which they are attached form a succinimide ring which is unsubstituted or substituted by alkyl having 1 to 12 carbon atoms, a maleimide, dimethylmaleimide, phthalimide, tetrahydrophthalimide or hexahydrophthalimide ring,

BSPR:

Preferred compounds of formula I are those wherein if n is 1 Y and R.sub.7 together with the N-atom to which they are attached form a succinimide, maleimide, dimethylmaleimide or phthalimide ring,

BSPR:

Synthetic resins that can have their resistance to deterioration enhanced with bis-(piperidyl) diimides according to this invention include .alpha.-olefin polymers such as polyethylene, polypropylene, polybutene, poly-3-methylbutene, or mixtures thereof and copolymers with other monomers such as ethylene-vinyl acetate copolymer; ethylene-propylene copolymer; polystyrene; polyvinyl acetate; polyacrylic esters; copolymers from styrene and another monomer (for example, maleic anhydride, butadiene, and acrylonitrile); acrylonitrile-butadiene-styrene copolymer, acrylic acid ester-butadiene-styrene copolymer, methacrylic acid ester-butadiene-styrene copolymer, polymethacrylate esters such as polymethacrylate; polyvinyl alcohol; polyvinyl formal; polyvinyl butyral; linear polyesters, polyamides; polycarbonates; polyacetals; polyurethanes, cellulosic resins; phenol-formaldehyde resins; urea-formaldehyde resins; melamine-formaldehyde resins; epoxy resins; unsaturated polyester resins; silicone resins; halogen-containing resins such as polyvinyl chloride, polyvinylidene chloride, polyvinylidene fluoride, and copolymers thereof, and rubbers such as isoprene rubber, butadiene rubber, epichlorohydrin rubber, chloroprene rubber, and blends of any of the above.

BSPR:

The stabilizer or combination is incorporated in the polymer in suitable mixing equipment, such as a mill or a Banbury mixer. If the polymer has a melt viscosity which is too high for the desired use, the polymer can be worked until its melt viscosity has been reduced to the desired range before addition of the stabilizer. Mixing is continued until the mixture is substantially uniform. The resulting composition is then removed from the mixing equipment and brought to the size and shape desired for marketing or use.

BSPR:

The polymer materials stabilized with the stabilizer of the invention can be any physical forms, including filaments, yarns, films, sheets, molded articles, latex, foams and coatings.

CLPR:

8. A stabilizer composition for synthetic resins comprising:

CLPR:

9. A polyvinyl chloride resin composition having improved resistance to deterioration comprising a polyvinyl chloride resin and bis-(2,2,6,6-tetramethyl piperidyl)diimide in accordance with claim 1.

unreacted isocyanato groups in the product. A final composition of the invention results if the compound used in step (2) contains only one isocyanato group or if all isocyanato groups of a di- or polyisocyanate are reacted, and if the compound used in step (2) contains a functional group that imparts soft hand, stain release, water repellency, or a durable property.

for example, the reaction of a fluorocarbon with the monoisocyanate affords a urethane as a compound in a final composition of the invention. Derivatives of this urethane, e.g., an allophanate, can also be formed in such a reaction and can also be present in a final composition of the invention. The reaction of a monoamine-functional fluorocarbon with a monoisocyanate affords a urea as a compound that can be present in a final composition of the invention. Derivatives of this urea, e.g., a biuret, can also be formed and can also be present in a composition of the invention. Also, condensation of two moles of an organic isocyanate with loss of  $\text{CO}_2$  affords a carbodiimide, which can then react with an amine-functional fluorocarbon to form a compound that can be present in a final composition of the invention.

In the case of di- and polysocyanates, the product of step (2) will be an intermediate compound of the invention if there remain unreacted isocyanato groups, or a final compound of the invention if all isocyanato groups have been reacted. As discussed above, urethanones, ureas, allophanates, biurets, guanidines, oxalourea derivatives, and sulfur analogs thereof can be formed.

Examples of isocyanates suitable for use in step (2) include monoisocyanates such as phenylisocyanate and octadecylisocyanate; aromatic disiocyanates such as 4,6-dinitrobenzoyl chloride diphenylene diisocyanate, 4-methylphenylenediisocyanate, 1,3-benzenedisocyanate, 2-toluenediol diisocyanate, o-, m-, and p-xylene diisocyanate, 4,4'-diisocyanatodiphenylmethane, 3,3'-dichloro-4,4'-diisocyanatodiphenylmethane, 4,5'-diphenyldiisocyanate, 4,4'-disocyanatodiphenylether,

1,3-dimethoxy-4,4'-disocyanato diphenyl, 2,2'-dichloro-5,5'-dimethyl-1,4'-disocyanatodiphenyl, 2,2'-dichloro-3,3'-dimethoxy-4,4'-disocyanatodiphenyl, 3,3'-dimethoxy-4,4'-disocyanatodiphenyl, 4,4'-disocyanatodiphenyl, 3,3'-dichloro-4,4'-disocyanatodiphenyl, 4,5'-dichloro-4,4'-disocyanatodiphenyl, 4,5'-dichloro-4,4'-disocyanatodiphenyl, 4,4'-disocyanatodiphenyl, 1,2-naphthylene disocyanate, 1,3-naphthylene disocyanate, 1,8-dinitro-2,7-naphthylene disocyanate, allylic disocyanates such as 3-isocyanatomethyl-3,5,5-trimethylcyclohexylisocyanate; aliphatic diisocyanates such as methylenedisocyanate, 1,6-hexamethylenedisocyanate, 2,2,4-trimethyl-1,6-hexamethylenedisocyanate, and 1,3-bis(isocyanatomethyl)benzene.

nitrocyanoate; aromatic polyisocyanates such as aliphatic triisocyanates such as 1,3,6-hexamethylene diisocyanate, and trimethylolpropane triisocyanate. In a product resulting from the use of such an isocyanate in step (2), the olefinic unsaturation can be removed by polymerization with another

...with a silicone compound, or by other conventional reactions.

CLPR:

10. A polyvinyl chloride resin composition in accordance with claim 9 in which the polyvinyl chloride resin is polyvinyl chloride homopolymer.

CLPR:

11. A polyvinyl chloride resin composition in accordance with claim 9 in which the polyvinyl chloride resin is a copolymer of vinyl chloride and vinyl acetate.

CLPR:

12. An olefin polymer composition having improved resistance to deterioration comprising an olefin polymer selected from the group consisting of polymers of alpha-olefins having from two to six carbon atoms and polystyrene, and bis-(2,2,6,6-tetramethyl piperidyl)diimide in accordance with claim 1.

CLPR:

13. An olefin polymer composition in accordance with claim 12 wherein the polyolefin is polypropylene.

CLPR:

14. An olefin polymer composition in accordance with claim 12 wherein the polyolefin is polyethylene.

CLPR:

15. An olefin polymer composition in accordance with claim 12 wherein the polyolefin is ethylene-propylene copolymer.

CLPR:

16. A polyurethane resin composition having improved resistance to deterioration comprising a polyurethane resin and bis-(2,2,6,6-tetramethyl piperidyl)diimide in accordance with claim 1.

blocking agent, e.g., an oxime such as aceton oxime, to afford the blocked isocyanato group.

Another method (not shown in the Scheme) of preparing a compound of this invention wherein the organic moiety R contains a blocked isocyanato involves (1) blocking the isocyanato group of an isocyanate-functional monomer such as isocyanatoethyl methacrylate to afford a blocked monomer; (2) oligomerizing the blocked monomer in the presence of an appropriate functional end-capping agent to provide a functional blocked oligomer containing blocked isocyanato groups and a functional group capable of reacting with an isocyanate; (3) reacting a deficiency of the functional oligomer with an organic di- or polyisocyanate to afford an isocyanato-containing intermediate compound also containing blocked isocyanato groups; and (4) reacting the remaining isocyanato groups of the product with a fluorochemical oligomer of Formula VII or VIII to form a linking group Z. In such a compound, the organic moiety R includes the blocked oligomer formed in step (2) with the organic nucleus of the di- or polyisocyanate used in step (3) bonded thereto through the isocyanate-derived linking group formed in step (3). Z is the isocyanate-derived linking group formed in step (4); and the remainder of the compound is as defined for Formula VII or VIII above.

Organic moiety R can contain other types of groups.

Such can be introduced in step (3) by, for example, reacting an isocyanate of Formulas IA, IIA, or IIIA with a reagent containing a group reactive to an isocyanate. When a reagent containing two or more reactive functional groups is used in such a reaction, at least one of the functional groups has a substantially lesser degree of reactivity with isocyanates than the others. The less reactive group will generally remain unreacted. Examples of reagents suitable for use in step (3) include acids such as lauric acid, palmitic acid, stearic acid, oleic acid, sorbic acid, limoleic acid, oxalic acid, succinic acid, adipic acid, benzotic acid, phthalic acid, terephthalic acid, fluorochemical alcohols, amines, and thiols such as those disclosed, for example, in U.S. Pat. Nos. 2,666,797 (Husted), 2,691,043 (Husted), 3,398,182 (Guenther et al.), and 4,606,737 (Stem) the disclosures of which are incorporated herein by reference; functionalized alcohols and amines such as glycidol, aziridine, 2-methylaziridine, 2-chloroethanol, 1,3-dichloropropanol, 2,3-dibromopropanol, and 1-chloro-2,3-propanediol; mono-, di-, or triols (such as those available as PEGCH<sub>2</sub>M, 3M), hydroxyacids such as malic acid, glyoxal, 12-hydroxydodecanoic acid, citric acid, tartaric acid, dimethylolacetic acid, dimethylolpropionic acid, p-hydroxybenzoic acid; aminoalcohols such as triethanolamine, N,N'-dimethylaminoethanol, methyltriethanolamine, ethyldiethanolamine, aminoaliphatic acids such as 4-(2-hydroxyethyl)-1-piperazineethanesulfonic acid, laurine, amino acids such as 6-aminocaproic acid; mono-, di-, or polyalcohols such as methanol, ethanol, butanol, 2-ethylhexanol, stearylalcohol, ethylene glycol, glycerol, 1,2,4-butanetriol, pentaerythritol, 1,6-hexanediol, 1,10-decanediol; mono-, di-, or polyamines such as butylamine, dibutylamine, diethylamine, ethylenediamine, 1,6-hexanediamine, diethylhexanetriamine, triethylhexanetriamine, tetra-

Also useful are isocyanates containing internal isocyanate-derived moieties such as biuret-containing triisocyanates such as that available from Mobay as DESMONDUR TM N-100, isocyanurate-containing triisocyanates such as that available from Huls AG, Germany, as IPDI-1890, and azetidinone-containing diisocyanates such as that available from Bayer as DESMONDUR TM TT. Also, other triisocyanates such as tri-(4-isocyanatophenyl)-methane (available from Bayer as SMONDUR TM TT). Also, other triisocyanates such as triisocyanate-containing moieties wherein the organic moiety R comprises a composition with phosgene and which will afford a triisocyanate containing mono-, di-, or polyisocyanate siloxane-con-

Other suitable types of isocyanates for use in step (2) and each Z will be a urethane group. The three isophoronyl substituents thereon, n will be 3; derivative isophoronyl nucleus of the IPDI-1890 and Formula I or III can be considered to be the isocyanate of the product of Formula VII, the organic moiety R in the product of Formula VII, and all isocyanato groups are reacted with a hydroxy-functional oligomeric intermediate of Step (2), and all isocyanato groups are reacted with a hydroxy-functional oligomeric intermediate of Step (2), and all isocyanato groups are reacted with a hydroxy-functional oligomeric intermediate of Step (2).

When, for example, IPDI-1890 is used as the isocyanate in Step (2), the reaction is run in the presence of a catalyst. Suitable catalysts include tertiary amines such as triethylamine, triethylamine, N-methylmorpholine, N,N,N',N'-tetramethyl ethylene diamine, N-methyldiethanolamine, N,N'-dialkylalkanolamines; tin salts such as dibutyltin dilaurate, stannous octoate, stannous oleate, tin dibutyltin (2-ethyl hexamose), stannous chloride, and others known to those skilled in the art. The preferred catalyst depends on the reactants (e.g., the nature of the nucleophilic functional groups in the fluorochemical oligomer and the aromatic or non-aromatic nature of the isocyanate) and on the particular isocyanate derivative that is the intended product of the reaction. For example, tin catalysts such as stannous octoate are preferred in the preparation of urethanes, while allophanate formation can be catalyzed by amines such as triethylamine. Urea formation can be carried out in the absence of catalyst. The amount of catalyst present will depend on the particular reaction, and thus it is not practical to recite particular preferred concentrations. Generally, however, suitable catalyst concentrations are from about 0.001 percent to about 10 percent, preferably about 0.1 percent to about 5 percent, by weight based on the total weight of the reactants.

Step (2) is preferably carried out under dry conditions in a polar solvent such as ethyl acetate, acetone, methyl isobutyl ketone, and the like. Suitable reaction temperatures will be easily determined by those skilled in the art based on the particular reagents, solvents, and catalysts being used. While it is not practical to enumerate particular temperatures suitable for all situations, generally suitable temperatures are between about room temperature and about 200° C.

In step (3) of Scheme I and II, an intermediate of Formula IA, IIA, or IIIA is reacted to form a compound of Formula I, II, or III respectively. As described above, the group R of a compound of Formula I, II, or III can contain blocked isocyanato groups. To prepare such compounds, an intermediate of Formula IA, IIA, or IIIA can be reacted with an isocyanate